## Bench-Scale Demonstration of Hot-Gas Desulfurization Technology

## **Quarterly Report**

October 1 - December 31, 1995

HEVEINED

FFR 1 4 1997

OSTI

Work Performed Under Contract No.: DE-AC21-93MC30010

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
Research Triangle Institute
P. O. Box 12194
Research Triangle Park, North Carolina 27709





#### DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

## Disclaimer

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

## **TABLE OF CONTENTS**

Sect	ion		Page
1.0	Introd	duction and Summary	1-1
2.0	Tech	nical Discussion	2-1
	2.1	Field Testing of ZTFBD/DSRP at METC - Data Analysis and Final Report Draft	2-1
	2.2	Scaled-Up DSRP Reactor System	2-12
	2.3	Technology Transfer	2-12
3.0	Plans	s for Next Quarter	3-1

#### 1.0 INTRODUCTION AND SUMMARY

The U.S. Department of Energy (DOE), Morgantown Energy Technology Center (METC), is sponsoring research in advanced methods for controlling contaminants in hot coal gasifier gas (coal gas) streams of integrated gasification combined-cycle (IGCC) power systems. The programs focus on hot-gas particulate removal and desulfurization technologies that match or nearly match the temperatures and pressures of the gasifier, cleanup system, and power generator. The work seeks to eliminate the need for expensive heat recovery equipment, reduce efficiency losses due to quenching, and minimize wastewater treatment costs.

Hot-gas desulfurization research has focused on regenerable mixed-metal oxide sorbents which can reduce the sulfur in coal gas to less than 20 ppmv and can be regenerated in a cyclic manner with air for multicycle operation. Zinc titanate (Zn<sub>2</sub>TiO<sub>4</sub> or ZnTiO<sub>3</sub>), formed by a solid-state reaction of zinc oxide (ZnO) and titanium dioxide (TiO<sub>2</sub>), is currently one of the leading sorbents. Overall chemical reactions with Zn<sub>2</sub>TiO<sub>4</sub> during the desulfurization (sulfidation)-regeneration cycle are shown below:

Sulfidation:  $Zn_2TiO_4 + 2H_2S \rightarrow 2ZnS + TiO_2 + 2H_2O$ 

Regeneration:  $2ZnS + TiO_2 + 3O_2 \rightarrow Zn_2TiO_4 + 2SO_2$ 

The sulfidation/regeneration cycle can be carried out in fixed-bed, moving-bed, or fluidized-bed reactor configuration, and all three types of reactors are slated for demonstration in the DOE Clean Coal Technology program. The fluidized-bed reactor configuration is most attractive because of several potential advantages including faster kinetics and the ability to handle the highly exothermic regeneration to produce a regeneration offgas containing a constant concentration of SO<sub>2</sub>. However, a durable

attrition-resistant sorbent in the 100- to 400-µm size range is needed for successful fluidized-bed operation.

The SO<sub>2</sub> in the regeneration offgas needs to be disposed of in an environmentally acceptable manner. Options for disposal include recycle to the gasifier in which an in-bed desulfurization sorbent such as dolomite or limestone is being employed, conversion to sulfuric acid, and conversion to elemental sulfur. All three options are being pursued and/or proposed in the Clean Coal Technology program. Elemental sulfur recovery is the most attractive option because sulfur can be easily transported, stored, or disposed. However, elemental sulfur recovery using conventional methods from an offgas containing low levels of SO<sub>2</sub> (typically 3%) is an expensive proposition. An efficient, cost-effective method is needed to convert the SO<sub>2</sub> in the regenerator offgas directly to elemental sulfur.

Research Triangle Institute (RTI) with DOE/METC sponsorship has been developing zinc titanate sorbent technology since 1986. In addition, RTI has been developing the Direct Sulfur Recovery Process (DSRP) with DOE/METC sponsorship since 1988. Fluidized-bed zinc titanate desulfurization coupled to the DSRP is currently the most advanced and attractive technology for sulfur removal/recovery for IGCC systems, and it has recently been proposed in a Clean Coal Technology project.

RTI has developed a durable fluidized-bed zinc titanate sorbent, ZT-4, which has shown excellent durability and reactivity over 100 cycles of testing at 750 to 780°C. In bench-scale development tests, it consistently reduced the H<sub>2</sub>S in simulated coal gas to <20 ppmv and demonstrated attrition resistance comparable to fluid cracking catalysts. The sorbent is manufactured by a commercially scalable granulation technique using commercial equipment available in sizes up to 1,000 L. The raw materials used are

relatively inexpensive, averaging about \$1.00/lb. It is anticipated that the impact on cost of electricity (COE) due to sorbent replacement for attrition will be less than 0.5 mil/kWh. ZT-4 has recently been tested independently by the Institute of Gas Technology (IGT) for Enviropower/Tampella Power, and showed no reduction in reactivity and capacity after 10 cycles of testing at 650°C.

In the DSRP SO<sub>2</sub> is catalytically reduced to elemental sulfur using a small slip stream of the coal gas at the pressure and temperature conditions of the regenerator offgas. A near-stoichiometric mixture of offgas and raw coal gas (2 to 1 mol ratio of reducing gas to SO<sub>2</sub>) reacts in the presence of a selective catalyst to produce elemental sulfur directly:

$$2H_2 + SO_2 \rightarrow (1/n)S_n + 2H_2O$$
  
 $2CO + SO_2 \rightarrow (1/n)S_n + 2CO_2$   
 $CO + H_2O \rightarrow CO_2 + H_2$ 

The above reactions occur in Stage I of the process, and convert up to 96% of the inlet SO<sub>2</sub> to elemental sulfur, which is recovered by cooling the outlet gas to condense out the sulfur. Adjusting the stoichiometric ratio of coal gas to regenerator offgas to 2 at the inlet of the first reactor also controls the Stage I effluent stoichiometry since any H<sub>2</sub>S and COS produced (by the reactions:  $3H_2 + SQ_2 - H_2S + 2H_2O_2$ ) yields an (H<sub>2</sub>S + COS) to  $SQ_2$  ratio of 2 to 1. The effluent stoichiometry plays an important role in the Stage II DSRP reactor (operated at 275 to 300°C), where 80% to 90% of the remaining sulfur species is converted to elemental sulfur most probably via COS + H<sub>2</sub>O - H<sub>2</sub>S + CQ and  $2H_2S + SQ_2 - (3/\eta_2)S_2 + 2H_2O_2$ . The overall sulfur recovery is projected at 99.5%.

The DSRP technology is also currently at the bench-scale development stage with a skid-mounted system ready for field testing. Very recently, the process has been extended to fluidized-bed operation in the Stage I reactor. Fluidized-bed operation has proved to be very successful with conversions up to 94% at space velocities ranging from 8,000 to 15,000 scc/cc·h. Overall conversion in the two stages following interstage sulfur and water removal has ranged up to 99%.

A preliminary economic study for a 100 MW plant in which the two-stage DSRP was compared to conventional processes indicated the economic attractiveness of the DSRP. For 1% to 3% sulfur coals the installation costs ranged from 25 to 40 \$/kW and the operating costs ranged from 1.5 to 2.7 mil/kWh.

Through bench-scale development, both fluidized-bed zinc titanate and Direct Sulfur Recovery Process (DSRP) technologies have been shown to be technically and economically attractive. The demonstrations to date, however, have only been conducted using simulated (rather than real) coal gas and simulated regeneration off-gas. Thus, the effect of trace contaminants in real coal gases on the sorbent and DSRP catalyst is currently unknown. Furthermore, the zinc titanate work to date has emphasized sorbent durability development rather than database development to permit design of large-scale reactors. Discussions with fluidized-bed experts have indicated that data from a larger reactor than the present are required for scaleup, especially if the material does not have particle sizes similar to fluid catalytic cracking catalysts (typically ~80 µm). The fluidized-bed zinc titanate technology uses 100- to 400-µm particles. Finally, the zinc titanate desulfurization unit and DSRP have not been demonstrated in an integrated manner.

The goal of this project is to continue further development of the zinc titanate desulfurization and DSRP technologies by

- Scaling up the zinc titanate reactor system;
- Developing an integrated skid-mounted zinc titanate desulfurization-DSRP reactor system;
- Testing the integrated system over an extended period with real coal-gas from an operating gasifier to quantify the degradative effect, if any, of the trace contaminants present in coal gas;
  - Developing an engineering database suitable for system scaleup; and
- Designing, fabricating and commissioning a larger DSRP reactor system capable of operating on a six-fold greater volume of gas than the DSRP reactor used in the bench-scale field test.

#### 2.0 TECHNICAL DISCUSSION

# 2.1 FIELD TESTING OF ZTFBD/DSRP AT METC - DATA ANALYSIS AND FINAL REPORT DRAFT

The following is a detailed description of the July, 1995, field test chronology. It has been drafted to be part of the final report for this project, and is included here to give the background on each of the designated "runs" listed in the table of results.

#### CHRONOLOGY OF FIELD TEST

On Monday, July 17, 1995, the METC gasifier started up on schedule, and in parallel RTI personnel heated up reactors and heat tracing in the RTI trailer in preparation for receiving coal gas. The initial operating strategy was to operate the DSRP for 160 hours continuously with coal gas feed and simulated regeneration off-gas (using vaporized liquid SO<sub>2</sub>). In parallel, the ZTFBD unit was to operate for 100 hours. Startup of both units was smooth, and after about 3 hours of operation, lined-out performance of the DSRP was achieved. Unfortunately, after the DSRP had been operating with coal gas for only 4 hours, the METC gasifier shut down. This initial period of operation was designated as **Run #1**.

Coal gas was available again on the morning of Tuesday, July 18, 1995. This next period of operation was designated as **Run #2**; the same operating parameters were chosen as were used for Run #1. During this period, the filter on the RTI end of the coal gas slipstream line started to plug up. The differential pressure transmitter across the filter went over range. Also, sulfur plugging in the sample line at the outlet of the DSRP was

noted, so that some of the analysis data are unreliable. A period of lined-out operation was achieved, however.

For **Run #3**, which was contiguous with Run #2 and started about noon on July 18, the DSRP reactor furnace set point was raised 20 °C. This caused the reaction temperature (the bottom catalyst bed temperature) to increase from 612 °C to 622 °C. The sulfur plugging in the low temperature DSRP outlet piping was first noticed during this run. The flow of liquid SO<sub>2</sub> was stopped briefly several times during this run in order to allow time for clearing plugs.

In the late afternoon of July 18, the DSRP reactor furnace temperature set point was lowered 40 °C to 580 °C and was designated the start of **Run #4**. This caused the reaction temperature (the bottom catalyst bed temperature) to decrease from 622 °C to 588 °C. During this run the plugging of the coal gas filter became more severe. The DSRP system pressure had to be reduced from 262 psig to 242 psig in order to maintain flow of coal gas into the unit. At midnight on July 18 the reactor furnace temperature was raised 20 °C back to the original set point, and the temperature experiment series ended.

The pressure drop across the coal gas filter continued to increase, so that **Run #5** (early morning of July 19) became a *de facto* reduced pressure run. The DSRP system pressure was reduced to 202 psig. Also, severe plugging of the outlet of DSRP was noticed. The coal gas flow to both the ZT unit and the DSRP was stopped so the equipment could be worked on to remove plugs in the DSRP outlet piping.

The morning of July 19 was spent clearing sulfur plugs from the outlet piping and devising a temporary piping arrangement that would permit back-pulsing of the coal gas (Mott) filter. At this point in the test program it became apparent that with continuous

operation of the DSRP with liquid SO<sub>2</sub> feed, the production rate of sulfur by the reactor system was overwhelming the capacity of the off-gas system (including the knock-out pot) to handle it. It was surmised that not all of the condensed sulfur was being removed from the gas stream by the separator pot. The presumed "mist" was then being vaporized in the reheater, passing through the back-pressure control valve as a vapor, and finally condensing elsewhere in the cool off-gas system either as a crystalline form or as a sublimed "flowers of sulfur" form. A decision was made to modify the operating strategy for the remainder of the test program.

The new operating plan was to run coal gas through the DSRP at all times in order to expose the catalyst for 160 hours. The nitrogen portion of the simulated regeneration off-gas would also be flowing through the DSRP reactor. The liquid SO<sub>2</sub> would only be run at selected times to observed how the catalyst was continuing to perform. The coal gas flow was restarted the afternoon of July 19 with the revised operating plan.

The coal gas filter back-pulsing procedure was followed for the first time, and it successfully reduced the pressure drop across the Mott filter from 75 psig to less than 10 psig. At this time the small Balston filters were removed from trace contaminant sampling points TCT-1 and TCT-2 (on the ZT unit). It was decided that gas from those sample points would not have large amounts of particulate that would clog the sampling apparatus and so would need to be filtered out in order to get reliability. Furthermore any small amount of particulate should preferably be collected as part of the sample in order to get a valid reading of the trace contaminants present.

Unfortunately, after several hours of coal gas and nitrogen flow, the DSRP outlet piping plugged up again. It appeared that residual sulfur in the off-gas lines was

"migrating" along and forming new plugs. Plugging of the lines outside the RTI trailer was also noted, and the critical flow orifice in the by-pass line was cleared by METC personnel. Coal gas flow was interrupted for several hours to permit line clearing efforts. A copper coil for cooling water was fabricated for installation in the knock-out pot

Coal gas flow was restarted the evening of the 19th, and continued to flow through the night. The flow was interrupted in the late morning in order to install the cooling coil inside the knock-out pot. Liquid SO<sub>2</sub> was not used again until the afternoon of July 20, the start of Run #6. There were a number of control problems encountered during this run. The pressure had to be continually decreased due to rising pressure drop across the coal gas filter. The output of the Western SO<sub>2</sub> analyzer appeared to be at odds with the reading on the liquid SO<sub>2</sub> rotameter. A brief experiment in which the automatic valve on the liquid SO<sub>2</sub> supply was closed showed an immediate effect on the rotameter but no effect on the Western reading. Only a very brief period of lined-out operation was achieved during this run. The liquid SO<sub>2</sub> flow was stopped in the late afternoon of July 20, and coal gas continued to flow.

A modification was made to the DSRP process equipment to add heat tracing to the liquid SO<sub>2</sub> feed line where it intersected the nitrogen line upstream of the preheater. This modification was expected to pre-vaporize the liquid and insure more complete mixing of the stream before the sample point for the Western SO<sub>2</sub> analyzer. The results were apparent during **Run #7** on the afternoon of July 21. The analyzer output was steady and consistent with the rotameter reading. Lined-out operation was achieved easily.

On July 22, the METC gasifier was shut down in order to effect a repair of the incinerator stack. Coal gas would not be available to the MGCR (and hence to the RTI

trailer) from that afternoon until the evening of July 25, 1995. At this time the test run of the ammonia decomposition catalyst in the ZT unit was ended. Also, it was decided to end the trace contaminant sampling program. The total staffing of the RTI trailer was reduced. The ZT and DSRP reactors were maintained hot with a small nitrogen purge.

During the outage some minor maintenance activities of the process equipment was accomplished. A stainless steel coil was installed in the knock-out pot, replacing the copper coil (which had corroded substantially). The Mott filter was replaced with a fresh spare. The used filter was dumped out and found to be plugged with a large quantity of what looked like pure carbon (soot).

In the early evening of Tuesday, July 25, 1995, coal gas flow was restored to the DSRP unit. Coal gas also flowed to the ZT unit (in order to maintain a sufficiently large coal gas flow through the slip stream line), although that reactor was not maintained at the high test temperature.

Early Wednesday morning the coal gas flow from the gasifier was interrupted once again and was not available until that evening. During this outage the mechanical back pressure regulators on the ZT unit were removed and replaced to correct a problem noted the previous day.

During this second half of the test program, the pressure drop across the replaced Mott filter was greater than the range of the DP transmitter, but unlike the first day of the test, no continued large increase was observed. The back-pulsing procedure was not used.

With the coal gas flow restored on the evening of July 26, Run #8 was started. A higher SO<sub>2</sub> concentration (3.6% compared to 2.5% typically for the previous runs) was

on the ZT reactor. Even though coal gas was not supposed to be flowing through this unit, the isolation valve was evidently allowing some flow, and the cooling reactor flange sprang a leak. The coal gas line was capped off to stop this problem.

#9A the goal was to achieve the best operation possible, with operating conditions the same as earlier in the run. This was achieved with an operating temperature of 620 °C, 250 psig, and 3.5% SO<sub>2</sub> in the simulated regen off-gas. For Run #9B the system pressure was raised to 265 psig -- the maximum that could be achieved given the pressure of the coal gas, and the observed pressure drop through the Mott coal gas filter. Lined-out operation was easily achieved.

For the final experiment of the 160-hour DSRP test run, **Run #9C** the nitrogen flow making up the simulated regen off-gas was reduced, thereby increasing the SO<sub>2</sub> concentration. A distinct increase in reactor temperature was noted. There was some difficulty getting the proper coal gas flow to line out the unit. During this time the liquid SO<sub>2</sub> in the supply tank was exhausted, so the DSRP runs were ended.

The METC gasifier continued to operate to conduct other tests, but early in the morning of July 29 the RTI process equipment stopped taking coal gas. Hot purging was followed by cool down and shutdown procedures.

#### <u>RESULTS</u>

#### Data Reduction:

The critical parameter used to judge the performance of the DSRP is the conversion of the incoming gaseous sulfur compounds to elemental sulfur. The conversions shown in this report are rigorous calculations based on gas concentrations, as obtained from the

continuous analyzers and gas chromatographs. The calculations take into account the incoming sulfur species in both the regeneration off-gas (sulfur dioxide) and the coal gas used as the reducing gas (hydrogen sulfide). Volume changes in the flow rates due to the formation of, and eventual condensation of, water are included. Specifically, the calculations are as follows:

The flow rate of nitrogen making up the synthetic regeneration off-gas was known from the electronic mass flow controller. The concentration of SO<sub>2</sub> in the mixture of nitrogen and vaporized liquid SO<sub>2</sub> was measured by a continuous SO<sub>2</sub> analyzer, so that the molar flow rate of SO<sub>2</sub> into the reactor could be calculated. The coal gas flow into the reactor was measured on a wet basis by the orifice flow meter used as part of the flow control instrumentation. The composition of the coal gas (H<sub>2</sub>S, H<sub>2</sub>, and CO) on a dry sample basis was measured by an on-line gas chromatograph/mass spectrometer operated by METC and located near the gasifier. The water content of the coal gas was determined gravimetrically from timed condensate samples, by METC (with confirming information from RTI condensate sampling). The wet basis coal gas composition was then calculated, and the molar flow rates of H<sub>2</sub>S, H<sub>2</sub>, CO and H<sub>2</sub>O were determined.

An RTI gas chromatograph was used to measure the sulfur species in the DSRP outlet gas stream (H<sub>2</sub>S, COS, and SO<sub>2</sub>) on a water-free basis. The flow rate of this stream was not measured directly, however. Rather, it was derived from the stoichiometry of the reactions that took place. For purposes of the flow rate calculations, complete reaction of the sulfur dioxide and the active components of coal gas was assumed. Thus, all the inlet sulfur dioxide disappears and all the moles of hydrogen in the coal gas are converted to the same number of moles of water. That water, plus the water coming in with the coal

gas, was condensed before the sample was analyzed for sulfur compounds. The CO in the coal gas is converted to CO<sub>2</sub> with no change in the number of moles. Thus, the dry basis outlet flow rate was calculated as the sum of the nitrogen flow in and the coal gas flow in, less the water in the coal gas and the water produced.

Knowing the dry basis total outlet flow rate, the individual sulfur species flow rates could be calculated from the GC concentrations. All inlet sulfur molecules (from the regeneration off-gas and from the coal gas) that were not still present in the outlet gas as one of the three measured species -- H<sub>2</sub>S, COS, and SO<sub>2</sub> -- were assumed to be converted to elemental sulfur. The percent conversion was thus calculated as inlet molar flows minus outlet molar flows divided by inlet molar flows.

The instrumentation in the RTI Mobile Laboratory also included an Ametek analyzer (operating on an ultraviolet photometric principle) for continuous, on-line measurement of H<sub>2</sub>S and SO<sub>2</sub> concentrations in the DSRP off-gas. This instrument provided continuous feedback to the operator to optimize the coal gas flow rate, but it did not accurately measure the absolute concentrations of the gaseous sulfur species in the outlet gas. Carbonyl sulfide (COS) is not detected by the Ametek unit; however, its presence interferes with an accurate measure of the H<sub>2</sub>S concentration. According to information from Ametek, the COS ppm value adds to the H<sub>2</sub>S ppm value according to this equation:

$$(H_2S)_{Ametek} = [H_2S] + [COS]/2$$

This relationship did not seem to be completely accurate, though, as it was not substantiated by the GC analyses of the same stream. The elemental sulfur yield can be calculated from the Ametek values; but since the total concentration of gaseous sulfur compounds in the off-gas is under reported (there is not a one to one correspondence

between COS and H<sub>2</sub>S concentrations), calculations based on Ametek data overstate the conversion to elemental sulfur.

#### **Summary of Results:**

Table 1 summarizes the conditions in each of the designated run periods, and reports the calculations of the conversion to elemental sulfur made according to the description above.

#### Parametric Studies:

Although the basic concept of the July 1995 run was to operate continuously at steady state, there were some opportunities to make small changes in some operating parameters to observe their effect on the DSRP reactions. Table I summarizes the operating conditions during all of the times that liquid SO<sub>2</sub> was being fed to the reaction system. The parameters that were changed to form a series of independent variables were as follows:

- Reactor catalyst bed temperature
- System pressure
- SO<sub>2</sub> concentration in the simulated regeneration off-gas

Examining the results, the apparent dependent variables that were measured were the following:

- % Conversion to elemental sulfur (when the coal gas flow was optimized to minimize the H<sub>2</sub>S and SO<sub>2</sub> content of the off-gas)
- COS concentration in the off-gas

During the operation of the various runs, it was noted that the COS concentration could not be affected by changing the coal gas flow rate. Therefore, some other variable of operation was influencing that value.

With three independent variables and two dependent variables, six combinations are possible. Figures 1 through 6 are plots of the results. In previous work, higher conversions were achieved with higher bed temperatures. In these runs, however, Figure 1 shows that the higher temperatures appeared to result in slightly lower conversions. There is a great deal of scatter in the data, though, and the range of temperatures covered is narrow. Probably no conclusion should be drawn about the effect of temperature.

In previous work, higher conversions were also achieved with higher system pressure. (FINAL REPORT WILL HAVE REFERENCES INSERTED HERE). Figure 2, reporting the data from the July 1995 runs, suggests that this conclusion held true. However, all but two data points were in a very narrow pressure range.

Figure 3 reports the effect of inlet SO<sub>2</sub> concentration on conversion. The data cover a good range of concentrations: from 1 to 5 percent. Most of the conversions to sulfur range from 97.5 to 98.3%. The three data points around 2.5% SO<sub>2</sub> that lie below this value were taken at lower pressures. Conversion at 5% inlet SO<sub>2</sub> concentration was also slightly lower, at 96.6%. This was the last run and during this run it was not clear if the conditions had been fully optimized.

Figure 4 reports the effect of catalyst bed temperature on COS concentration in the off-gas. An apparent increase in COS formation with higher temperature is observed. However, it should be noted that reactor temperature is not entirely an isolated, independent variable. Figure 7 shows the relationship of reactor temperature to inlet SO<sub>2</sub>

concentration, a variable suspected of influencing COS formation. It can be seen that the higher reactor temperatures are associated with higher SO<sub>2</sub> concentrations.

Figure 5 reports the effect of system pressure on COS concentration in the off-gas.

The data seem widely scattered. Thus, there does not appear to be an effect of pressure on COS concentration, at least over the narrow range of pressures studied.

Finally, Figure 6 reports the effect of inlet SO<sub>2</sub> concentration on COS concentration in the off-gas. This is the clearest trend observed in this series of parametric studies, with COS increasing with increasing SO<sub>2</sub>. This trend is consistent with the understanding of the chemistry of the DSRP where COS is produced from the reaction of SO<sub>2</sub> with CO. Very little steam was present in the gas mixture inlet to the DSRP. As has been predicted previously by experimental and modeling methods (FINAL REPORT WILL HAVE REFERENCES INSERTED HERE), it is believed that the presence of more steam will increase the degree of the shift reaction, thereby increasing hydrogen, increasing sulfur conversion, and reducing COS formation.

#### 2.2 SCALED-UP DSRP REACTOR SYSTEM

Only a small amount of effort was expended in this quarter with respect to the large scale DSRP system. Furnace fabrication (at the vendor) continues; delivery is scheduled for end of January, 1996.

#### 2.3 TECHNOLOGY TRANSFER

A meeting was held at METC with representatives from the M.W. Kellogg Technology Company. The objectives of the meeting were to discuss RTI's and METC's sorbent development activities (discussed elsewhere), and to discuss the possible application of DSRP to future Kellogg activities. The emphasis was on IGCC applications, with some discussion for opportunities in petroleum refining.

### **3.0 PLANS FOR NEXT QUARTER**

- Start mechanical construction of the scaled-up DSRP system following receipt of furnaces.
- 2. Continue drafting the final report.

11.5666o-d.qtr

Figure 1. Effect of Catalyst Bed Temperature on Yield of **Elemental Sulfur** 

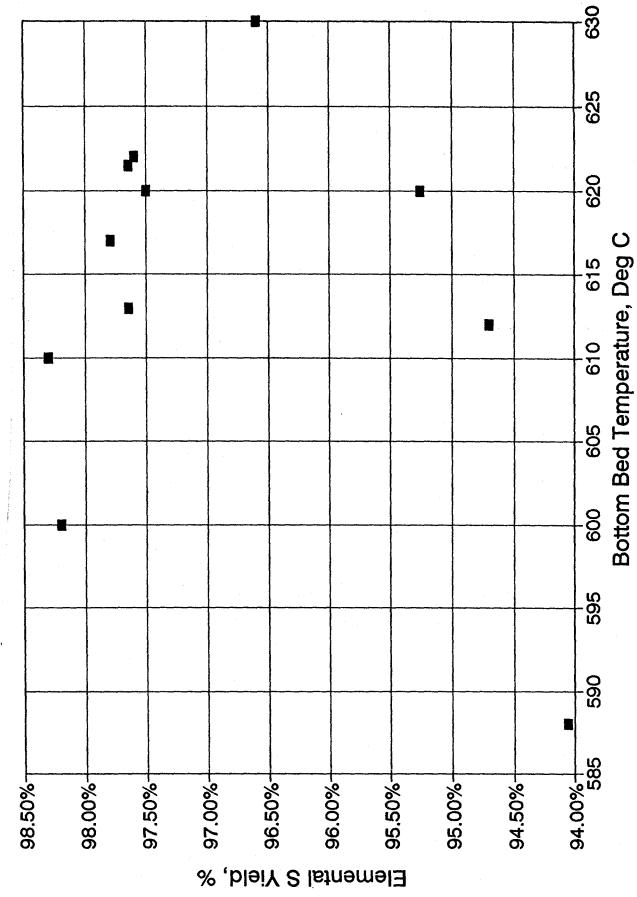
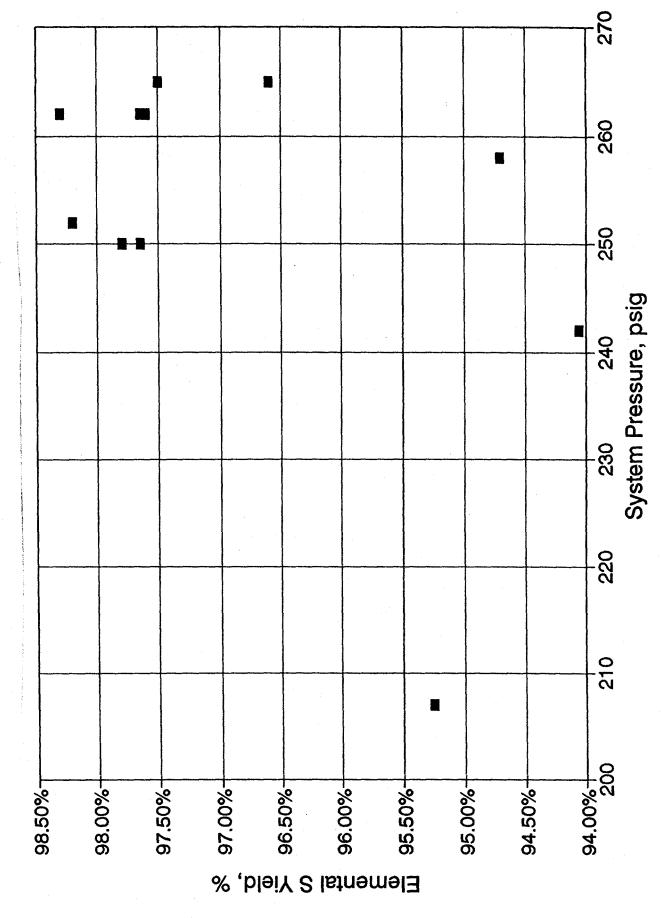


Figure 2. Effect of System Pressure on Yield of Elemental Sulfur



5.00% 4.50% Effect of Inlet SO<sub>2</sub> Concentrate on Yield of Elemental 4.00% 3.50% Inlet SO2 Conc'n, vol % 3.00% 2.50% 2.00% Sulfur Figure 3. 1.50% 94.00% -%00.86 97.00%-96.50%--%00'96 95.50%-95.00%-97.50%-98.50%-94.50%-Elemental S Yield, %

Bottom Bed Temperature, Deg C Concentration 585 300-700--009 Outlet COS Conc'n, ppm

Effect of Catalyst Bed Temperature on Outlet COS

Figure 4.

270 260 Figure 5. Effect of System Pressure on Outlet COS Concentration 250 System Pressure, psig 240 230 220 210 <del>|</del> 80 100 7007 -009 800 500-400 300-200-Outlet COS Conc'n, ppm

5.00% 4.50% 4.00% 3.50% Inlet SO2 Conc'n, vol % 2.50% 3.00% 2.00% Concentration 1.50% 1.00% 0.50% 0.00% 100 700 -009 500-400-300-200-800-Outlet COS Conc'n, ppm

Effect of Inlet SO<sub>2</sub> Concentration on Outlet COS

Figure 6.

630 625 Figure 7. Relationship of Inlet SO<sub>2</sub> Concentration to Catalyst Bed 620 Bottom Bed Temperature, Deg C 615 610 **Temperature** 900 595 590 1.00% 4.00% 2.00% 1.50% 3.00% 2.50% 5.00%-4.50%-3.50%-Inlet SO2 Conc'n, vol %

TABLE 1 - SUMMARY OF JULY 1995 DSRP TEST RUNS

		RUN	RUN	RUN	RUN	RUN #5	RUN #6	RUN	RUN		RUN #9	
		¥	#5	#3	#			<b>2</b> #	<b>*</b>	٧	8	ပ
Date		7/17/95	7/18/95	7/18/95	7/18/95	7/18-7/19	7/20/95	7/21/95	7/26/95	7/28/95	7/28-7/29	7/29/95
Start Time	me	18:25	05:40	12:17	17:11	23:45	13:13	14:15	23:35	21:00	23:37	00:19
End Time	ne	22:25	12:17	17:11	23:45	06:25	17:55	17:09	04:30	23:37	00:19	01:43
Duration, hr, w/LSO <sub>2</sub> on	n, hr, on	4.0	6.40	3.82	6.57	5.30	4.70	2.90	5.0		4.75	
Cum. tir of run	Cum. time @ end of run	4.0	10.57	15.68	22.35	28.92	52.64	76.40	112.1	153.5	154.1	155.6
RXTR Temp	Top TE-204	602	601- 603	613	929	609	596	601	607	605	909	605
ပ္	Middle TE-203	610	611- 613	621	584	616	605	611	616	614	615	616
	Bottom TE-202	610	612- 614	622	588	620	009	612	620- 623	617	620	930
System psig	System Pressure, psig	262	262	262	262- 242	242-202 (212-202)*	260-250 (252) <sup>b</sup>	258	250	250	265	265
SO <sub>2</sub> conc'n in Sim. ROG, %	nc'n in OG, %	3.1-3.2	2.8	2.5-2.7	2.5-2.7	2.8	4.0°	2.44	3.6	3.4-3.5	3.56	4.9
LSO, Re reading,	LSO, Rotameter reading, cc/min	6:0	0.9-1.0	1.0	1.0	1.0	0.52	6:0	1.4	1.4	1.4	1.4
Coal gas flow, SLPM	is flow,	11.9- 12.1	11.8- 13.6	11.3-	10.4- 16.6	9.7-12.7	7.5-8.4	11.2- 12.0	15.8- 16.1	15.4- 16.1	15.6-16.2	14-16
၀၁ လ	COS conc'n, ppm	29 <del>4</del> - 322	388- 444	395- 412	477- 537	510-542	180-243	360- 380	680- 715	570-610	616-650	670-815
% Conv Element	% Conversion to Elemental Sulfur	99.9 <b>-</b> 98.5	97.5- 98.0	97.6	93.4- 94.7	95.1-95.4	97.7- 98.7°	94.7	97.5- 97.8	97.6- 98.0	97.5	96.6 (avg'd)

- a. System pressure when lined-out.
- b. System pressure when lined out.
- c. Probably false high value from SO<sub>2</sub> analyzer. Based on liquid SO<sub>2</sub> rotameter, the concentration was probably actually about 1.4%.
- d. Additional heat tape added to liquid  $SO_2$  line for this and subsequent runs.  $SO_2$  analyzer values should be more reliable.
- e. Calculated percent reduction of sulfur compounds is 96% based on inlet SO<sub>2</sub> concentration of 1.4%.